УДК 621.039.66

# RADIATION RESISTANCE OF QUARTZ GLASSES

***B.A. Levin, D.V. Orlinski, K.Yu. Vukolov***
***RRC, Kurchatov Institute, Moscow 123182, RF***
***V.T. Gritsyna,***
***Kharkiv National University, Kharkiv 61077, Ukraine)***

The results of investigations of the quartz window radiation resistance for nuclear fusion reactor conditions are described. The tests of a few selected materials have shown that the best for a visible spectrum region is quartz KU-1 with a large hydroxyl (OH) content. In comparison with other tested types of quartz it has the lowest loss of the transparency, the lowest radioluminescence intensity and in comparison with other materials (sapphire, cerium glass and spinel) preserved its properties in a widest part of the spectrum after neutron, electron and gamma irradiation. Other type of the quartz glass with a small content of Cl was tested and found to be more suitable for UV spectral region.

## INTRODUCTION

Despite of numerous previous investigations [1] a problem of the transparent material choice of diagnostic windows for fusion reactor conditions needs in additional study. Reasons for this are: (1). The expected neutron fluence for windows in diagnostic channels may reach of $10^{18}…10^{19}$ n/cm$^2$ at ionization dose of 0.1-1 GGy(Si) (below all dose units are given for Si) (2). Fusion reactor radiation conditions (the values of neutron and gamma fluxes and the ratio between these fluxes) will differ from that of all other radiation sources. (3). The choice must be done from some materials of different characteristics.

Therefore the next main problems have to be solved: (i) the choice of the best materials from those that traditionally are used in thermonuclear devices. (ii) Determination of the dependencies of the transparency (or optical density) of the chosen material on fluxes, absorbed doses and type of irradiation. (iii) Radioluminescence measurement of material and compare it's intensity with the plasma visible radiation. (iv) Measurements of the transparency and radioluminescence dependencies on the temperature, and investigation of a possibility of material characteristics restoring by thermal annealing.

Below the results of investigations in this subject, which were accomplished in European, Japanese and Russian laboratories, are described.

## THE CHOICE OF THE WINDOW MATERIAL

The next materials were selected for preliminary tests: three types of the quartz glass with different content of hydroxyl – KUVI-S (~$10^{-4}$ % OH), KUVI-M (~0.01% OH) and KU-1 (~0.1% OH); sapphire, magnesium aluminate spinel $MgAl_2O_4$ and radiation resistant cerium glass S-96 [2]. Recently the transparency of quartz glass KS-4V [3] in a near UV region was studied and it radiation resistance was compared with that for other materials. Samples from all these materials usually were of 2 cm in diameter and 0.8 cm in thickness.

The next radiation sources were used for tests [2]: $^{60}$Co gamma source (dose rate $dD_\gamma/dt \leq 12$ Gy(Si)/s and dose $D_\gamma \leq 12$ MGy), pulse electron accelerators LU-40 in Kharkiv (pulse duration $\tau$=2.9 µs, repetition frequency f=12.5…150 Hz electron energy $E_e$=10 MeV, average $dD_\gamma/dt$ = 0.15…1.0 kGy/s, $D_\gamma \leq 8$ MGy), LU-50 in Sarov ($\tau$=10 ns, f ≤ 2.4 kHz, $E_e$=50 MeV, average $dD_\gamma/dt \leq 30$ Gy/s, $D_\gamma \leq 700$ kGy) and Van de Graaf electron accelerator in CIEMAT (0.7 kGy/s, $D_\gamma \leq 20$ MGy); neutron generators in Moscow region and in Japan (neutron flux on samples $dF_n/dt \approx 10^{11}$ n/cm$^2$s at $dD_\gamma/dt \approx 0.03…0.15$ Gy/s); nuclear pool-type reactors in Kurchatov Institute (for $E_n > 100$ keV, $dF_n/dt \approx 7 \cdot 10^{12}$ n/cm$^2$s $D_\gamma \leq 2.5$ MGy, $F_n \leq 10^{20}$ n/cm$^2$). Usually the temperature of sample during and after irradiation was not more than 30…50°C (in some experiments up to 300°C).

The main results of the tests were following: (1). Transparency of all tested materials is very sensitive to all kinds of the radiation in a near ultraviolet spectral region; (2). Spinel and cerium glass completely lost transparency at the wavelength region $\lambda \leq 400$ nm and were excluded from further tests, but their transparencies at $\lambda > 400$ nm are almost not sensitive to irradiation; (3). Sapphire also is very sensitive in all visible region (Fig.1) and may be used only for an infrared radiation [4]; (4).

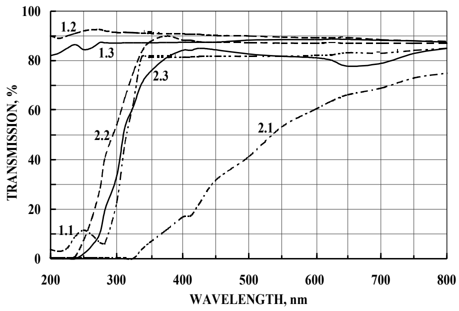


*Fig.1. Transmission spectra of sapphire (curves 1.1 and 2.1), quartz glass KU-1 (1.2 and 2.2) and quartz glass KUVI-S (1.3 and 2.3) before (1.1, 1.2 and 1.3) and after (2.1, 2.2 and 2.3) irradiation in the nuclear reactor to neutron fluence of $10^{18}$ n/cm$^2$ and $\gamma$-dose of 45 MGy [4]*

The difference in the transparency spectra for three types of quartz glasses is rather small and all they were left for additional tests.

## DEPENDENCE OF QUARTZ GLASS TRANSPARENCY ON IRRADIATION CONDITIONS

Irradiation of KU-1 samples in the nuclear reactor up to neutron fluence of $6x10^{19}$ n/cm$^2$ (total ionization dose of ~3 GGy) and by electron beams up to 10 MGy have shown that the optical density {OD=$\Delta^{-1}$lg($I_0$/I), were $\Delta$ is the sample thickness and $I_0$ and I are intensities of the incident and transmitted light} at $\lambda$>350 nm practically did not changed with exception of a week absorption band at $\lambda\approx$620 nm [5]. From the irradiation start OD at $\lambda$<350 nm increases gradually after the dose of ~10 kGy and then, from ~1 MGy, either saturates or even starts to decrease (Fig.2). After the termination of irradiation the optical density for $\lambda$<350 nm decreases sharply and in the short wavelength region optical density, measured after irradiation [6,7], shows less values than measured *in situ.*

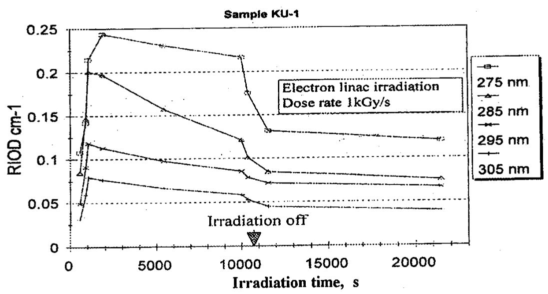


*Fig.2. Radiation Induced optical density (RIOD≡δ A=difference OD, measured during and before irradiations) for several wavelength, measured in situ under electron irradiation at temperature of ~70°C [7]*

Analogous results were obtained for the transparency dependence of glass on the neutron flux and gamma dose rate (Fig.3): both of them practically does not influence on the induced optical density at the wavelength region of $\lambda$> 400 nm, but at shorter $\lambda$ the larger flux leads to the lower transparency at equal fluences.

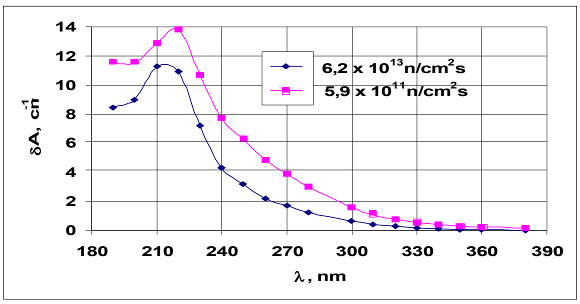


*Fig.3. Radiation induced optical density $\delta A(\lambda)$ of two KU-1 samples after irradiation to neutron fluence of $10^{17}$ n/cm$^2$ ($D_\gamma$~ 3 MGy) at different neutron fluxes - $5.9x10^{11}$ n/cm$^2$s and $6.2\cdot10^{13}$ n/cm$^2$s [5]*

The KU-1 optical density depends on the temperature during irradiation only at $\lambda$< 350 nm [8-10]. The higher is the sample temperature the faster increased the optical density during irradiation and the lower its saturation value at equal gamma doses and dose rates. The last effect corresponds to the possibility to restore the quartz transparency by heating. The direct measurements have shown that the transparency of irradiated to neutron fluence of (0.1…6)$\cdot10^{19}$ n/cm$^2$ KU-1 samples really are restored after heating up to 700…800°C [11].

At the expected reactor conditions between operations pulses the windows will be exposed to gamma irradiation from activated by neutrons reactor environment equipments. To check this effect the KU-1 sample, irradiated in a nuclear reactor up to fluence $10^{17}$ n/cm$^2$ was $\gamma$ irradiated to the additional dose of 3 MGy [12]. Its transparency in UV region almost was not changed.

## RADIOLUMINESCENCE OF QUARTZ GLASS KU-1

To be sure that the luminescence of the chosen window material under neutron and gamma irradiation will not prevent any optical measurements, the luminescence of some chosen materials under neutron, electron and gamma radiation was studied. The main difficulties of such experiments consist in requirement of measurements *in situ* at rather low luminescence intensity.

For *in situ* measurements the radiation source must assure the test of samples without introducing addition optical elements. This requirement excludes the use of stationary nuclear reactors. For low luminescence intensity measurement it is necessary to have very sensitive registration system or high intensity of radiation source. In these experiments the $^{60}$Co gamma source and neutron generator were used with sensitive detection system [13, 14]. The electron beam of Van de Graaf accelerator with dose rate of 700 Gy/s [15] and pulse nuclear reactor with an average dose rate for one pulse of 155 kGy/s was used also [16].

To compare luminescence of different materials the samples of sapphire, anhydroguide quartz (analog of KUVI-S) and KU-1 were tested under electron beam [15]. Anhydroguide quartz emission has pronounced maximum at $\lambda\approx$450 nm (this maximum was observed also under X-ray irradiation [8]) and low intensity maximum at $\lambda\approx$650 nm. Emission spectra of KU-1 glass have very smooth curve weakly pronounced maximum at 450 nm and 650 nm.

Common feature for both glasses is the increased emission at $\lambda\leq$300 nm, which decreases with neutron fluence and $\gamma$ dose due to increasing of the reabsorption by optical centers in this region. Results of absolute measurements of the radioluminescence (RL) intensity for tested materials are shown in Fig.4.

Measurements at different temperature T have shown that KU-1 luminescence does not depend on T at all. Emission of anhydroguide quartz at ~450 nm decreased with T and maximum disappears at ~350°C and became almost the same as KU-1 spectrum. Spectra both of them, corrected for the self-absorption, are near to the theoretical dependence of Cherenkov radiation on the wavelength ($\lambda^{-2}$).

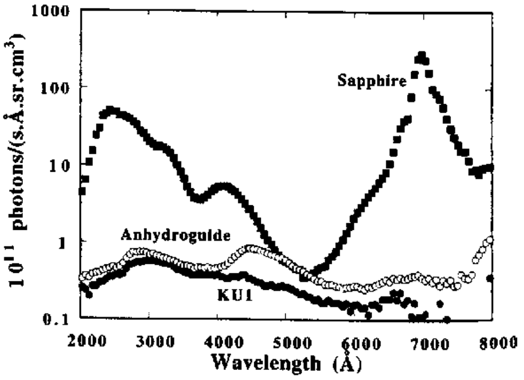


*Fig.4. Radioluminescence (RL) spectra for two types of $SiO_2$ and the Union Carbide UV grade sapphire after 2 h irradiation with the electron beam at 15°C, 700 Gy/s, and ~$10^{-10}$ dpa/s [15]*

Two identical samples of anhydroguide $SiO_2$ were irradiated from D-T neutron generator and from $^{60}Co$ gamma source. Results of luminescence measurement are shown in Fig.5 [13]. Data given in [13,14] allows to compare luminescence efficiencies for different materials under irradiation with neutrons from the D-T neutron generator and with gamma quanta from the $^{60}Co$ source.

Results of relative luminescence intensity measurements in the spectral region of 350…650 nm are adduced in Table 1 [13]. These data show that from the point of view of minimum radioluminescence background intensity in a visible spectral region the best materials are silica glasses and the best of these - the quartz glass KU-1.

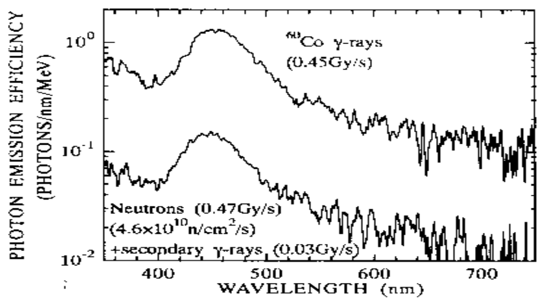


*Fig.5. Wavelength spectra of photons emitted by identical high-purity $SiO_2$ samples in DT-neutron and $^{60}Co$ gamma ray irradiation experiments at room temperature [13]*

**Table 1**

**Number of emitted photons per unit absorbed energy in a visible range (350…650 nm)**

| Sample | Peak wavelength (nm) | D-T neutrons and γ-rays (photons/MeV) | $^{60}Co$ γ-rays (photons/MeV) |
|---|---|---|---|
| Anhydroguide $SiO_2$ | 450 | 17±6 | 170±60 |
| KU-1 quartz glass | 650 | 14±5 | - |
| Ge doped silica | 390 | 83±30 | 410±140 |
| Sapphire | 410 | 2500±1000 | 27000±11000 |
| Calcium fluoride | <350, 550 | 270±110 | 1300±500 |

Radioluminescence measurements using pulse nuclear reactor have some advantages to compare with other sources: ratio of neutron to gamma fluxes is near to expected for the fusion reactor and pulse mode of irradiation allows to avoid of possible influence of the absorbed dose and also to measure in one pulse luminescence dependence on irradiation flux. Luminescence spectra for this case are shown in Fig.6.

Investigation of the temperature and preliminary irradiation influence have shown that temperature increase from 18 to 100°C does not changed luminescence intensity and after preliminary irradiation up to $6x10^{18}$ n/cm$^2$ the emission intensity at $\lambda \approx 620…670$ nm became almost 2.5 times larger, but after further increase neutron fluence up to $6 \cdot 10^{19}$ n/cm$^2$ intensity decreased by ~20%.

Other part of the spectrum remains unchanged. Results of KU-1 radioluminescence measurement at different radiation sources are given in Table 2. Comparison of estimated values of plasma continuum emission and windows radioluminescence for expected ITER conditions has shown [16, 17] that luminescence of KU-1 and anhydroguide quartz glasses will not prevent signal from plasma diagnostic but sapphire emission is near the brink permissible.

**Table 2**

**Radioluminescence intensity in the spectral region of 350…650 nm of KU-1 samples irradiated from different radiation sources**

| Radiation Source | Operating condition | Ionizing dose rate, (Gy/s) | Displacement dose rate, (dpa/s) | Photons number per 1 MeV of total absorbed energy | Ratio of radiated energy to absorbed energy |
|---|---|---|---|---|---|
| Electron accelerator | stationary | 700 | ~$10^{-10}$ | 80*) | ~ $2 \cdot 10^{-4}$ |
| Neutron generator | stationary | 0.5 | ~$10^{-10}$ | 14*) | ~ $4 \cdot 10^{-5}$ |
| Nuclear | pulse, FWHM | in one pulse at an | | | |

| reactor | 60μs | average155,000 | ~4·10^-5 | 4*) | ~ 1·10^-5 |
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*) Measurement accuracy 30…50 %

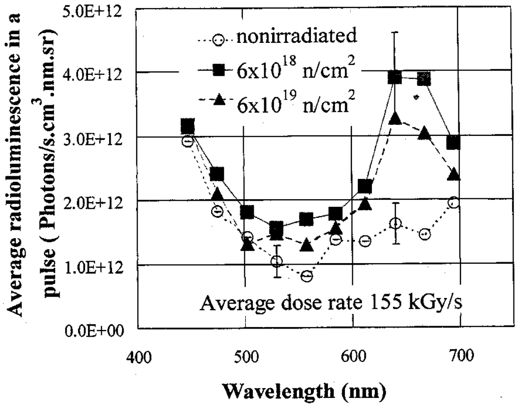


*Fig.6. Radioluminescence spectra of specimen KU-1, measured in a pulse nuclear reactor before and after preliminary irradiation in a fission reactor to two different fluences [16]*

All results of KU-1 tests have shown that this type of glass may be used for diagnostic windows in spectral region of λ>350 nm at expected for ITER radiation conditions.

## PROPERTIES OF QUARTZ GLASS KS-4V FOR UV REGION

Quartz glass KS-4V, manufactured in St-Petersburg (IRUVISIL Co., Ltd.), differs from other types of glasses by low content of Cl - 20 ppm (KU-1 - ~100 ppm) [3]. It has also very low content of hydroxyl (<0.1 ppm) and different metal impurities (3-15 times less than that in KU-1) KS-4V was recommended by manufacturer as radiation resistant in UV spectral region. Transparency spectra for this region of some samples from different ingots are shown in Fig.7 together with data for KU-1.

It could be seen that at λ>330 nm all samples KS-4V have practically the same transparency, but at the shorter wavelengths their transparencies are essentially different. There are some suppositions on the reason of such effect. The ingots of KS-4V differ not only in time and technology of their fabrication (after producing of ingot 5/155 a technology was changed) but in a content of metal impurities also. Transparency behavior of different samples to some extent correlates with an iron content, but opposite to the content change of aluminum. The content of other impurities in these samples is identical [18].

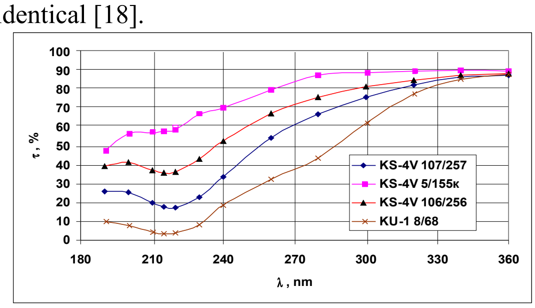


*Fig.7. Transparency spectra of samples KS-4V (thickness of 10 mm) from various ingots and KU-1 (thickness of 8 mm) one day after irradiation up to 900 kGy at 35°C*

At γ-doses of $D_\gamma \leq 270$ kGy short-living component of absorption for some samples was observed in a visible region [18]. If KU-1 optical density diminished only in UV spectral region (Fig.2) independently on gamma dose and dose rate [6], optical density of KS-4V decreased also in a visible region, but only at relatively small gamma dose (Fig.8).

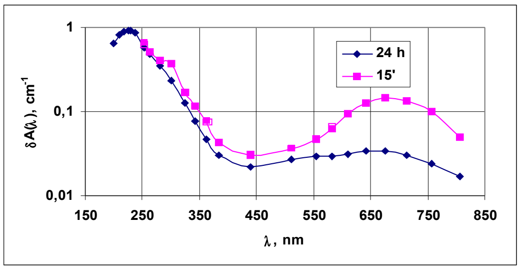


*Fig.8. Radiation induced optical absorption of KS-4V measured 15 minutes and 24 hours after irradiation at $D_\gamma$ = 9 kGy at 35°C [18]*

Much higher absorption was observed after irradiation in a nuclear reactor (Fig.9). In spite of KS-4V looks much more transparent than KU-1 their transparencies became <10% (δA=1 cm$^{-1}$) at λ<260 nm for KS-4V and at λ<290 nm for KU-1. For plasma diagnostics it does not make any difference.

As to KS-4V radioluminescence, it is typical for anhydroguide quartz glasses [20]: its spectra are looks like for KU-1 under neutron irradiation and have rather

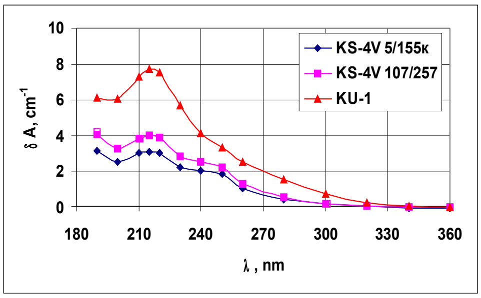


*Fig.9. Radiation induced absorption in UV region of KS-4V and KU-1 after irradiation in a nuclear reactor up to fluence of 3·10$^{16}$ n/cm$^2$ and γ-dose of ~1 MGy at the temperature of 16…30°C [19]*

large peak at ~450 nm under gamma or electron irradiations (Fig.10).

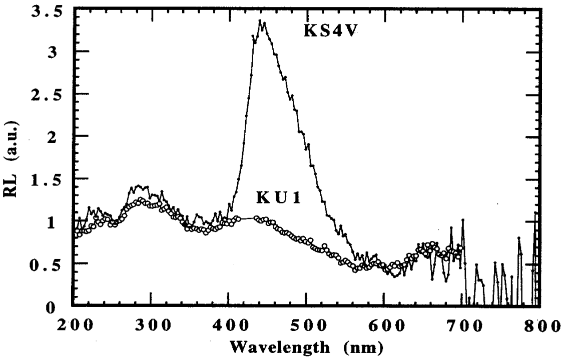


*Fig.10. Radioluminescence spectra of quartz glasses KU-1 and KS-4V under electron beam radiation at dose rate of 700 Gy/s and normal temperature [20]*

Comparative study of KS-4V and KU-1 characteristics has shown that at gamma and electron irradiation KS-4V has better transparency in UV region but much larger radioluminescence intensity then KU-1 glass. At neutron irradiation these glasses have almost identical characteristics.

## DISCUSSION

Adduced above data shows that all tested materials have large induced absorption coefficients mostly in the UV-region of spectra. The best materials are quartz glasses, but all of them have absorption bands at 215 and 260 nm. The 215 nm band is observed in all quartz glasses and is caused by E' centers, that is formed by trapping a radiolitic hole in Si-Si bond at the site of oxygen vacancy. The 260 nm band is more intense for OH-free quartz glass (Fig.9). It has been attributed to oxygen-related defects, such as non-bridging oxygen hole centers (NBOHC). Comparison of measured influence of reactor's neutron and electron irradiation [2,8] on optical absorption leads to conclusion: as a whole quartz glass, which has the high concentration of OH group (wet glass), is a little bit more radiation resistant to compare with dry glass (Fig.9).

From the spectra of RL under electron excitation one can distinguish bands in $SiO_2$ at 290, 450 and 650 nm [8]. The nature of indicated bands was discussed in numerous review papers. Most probable the 650 nm band is related to NBOHC, the 450 nm is related to the oxygen-deficiency centers (ODCs) - specifically to di-coordinated silicon -ODC(II). The band at 290 nm also related to ODC(II) - oxygen di-vacancy. It was found that relative RL intensities of bands very sensitive to the source of irradiation [14,15]. Under electron excitation at $dD_e/dt = 700$ Gy/s the intensities of different bands are comparable. In DT-neutron experiments (Fig.5) $dD_\gamma/dt = 0.03$ Gy/s the intensity of the 450 nm band is the lowest despite the simultaneous irradiation with neutrons at the $dF_n/dt = 4.6\cdot10^{10}$ n/cm$^2$s. Under irradiation only in $^{60}$Co gamma ray source at higher excitation ($dD_\gamma/dt=0.47$ Gy/s) the intensity of this band increases by the order of magnitude. Thus, most of this luminescence comes from the defects (either intrinsic or newly produced) in electron excitation. From Table 1 one can see that in DT experiments still the number emitted photons per unit absorbed energy is lowest for $SiO_2$ samples to compare with other potentially used materials for optical windows.

Comparison of the dose rate dependencies of optical properties of the KU-1 glass irradiated with pulse electron beam and continuous neutron bombardment indicates that formation of the stable optical centers involves the transient processes, that are dependent on the type of excitation and density of deposited energy.

The pulsed dose rate at the electron irradiation leads to the instant energy deposition of 2.2 MGy/s [8] in material that leads to creation the high density of defects. Post irradiation evolution and interaction between these transient defects facilitate creation of the stable optical centers. On the other hand, the continuous irradiation even at the highest neutron dose rate of $6.6\cdot10^{13}$ n/cm$^2$s produces transient defects with large spatial separation excluding their mutual interaction and nonlinear effect of stable center formation.

## CONCLUSION

These investigations were undertaken with the aim to find and to test a suitable material for windows of ITER diagnostic systems. Obtained data have shown that in ITER radiation conditions all types of quartz glass may be used in the spectral region of 350-2000 nm. A little bit better for this region is quartz KU-1 with a large content of hydroxyl – it can stand at the higher neutron fluence and has slightly lower radioluminescence intensity than anhydroguide, high purity and extremely purified (with small amount of Cl impurity) quartz. But the quartz glass KS-4V (of five times less content of chlorine) has in a near UV spectral region somewhat better transparency at neutron irradiation and much better one at gamma radiation dose of 1-3 MGy.

At the same time there are some questions, which are left without answers. (i). Almost all data on optical density were obtained after irradiation. Optical density in the short wavelength region during and after irradiation may be different. (ii). The ratio between neutron and gamma irradiation expected for a fusion reactor will be different from this one used in described experiments.

It was shown that effects of neutron and gamma irradiation are not identical. But it is unlikely that all of these circumstances can change the main results of presented studies – quartz glass KU-1 is the most radiation resistant material for diagnostic windows in a visible spectral region and KS-4V is better for optical radiation in the near UV range.

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## ДОСЛІДЖЕННЯ РАДІАЦІЙНОЇ СТІЙКОСТІ КВАРЦЕВИХ СТЕКОЛ ДО НЕЙТРОННОГО ТА ГАММА-ОПРОМІНЕННЯ

***Б.А. Левін, Д.В. Орлинський, К.Ю. Вуколов, В.Т. Гріцина***

Описані результати досліджень радіаційної стійкості деяких видів кварцових вікон для очікуваних умов термоядерного реактора. Випробування декількох вибраних матеріалів показали, що кварц КУ-1 з високим вмістом гідроксилу ОН кращий із досліджених для видимої області спектру. В порівнянні з другими зразками матеріалів (сапфір, церієве стекло та шпінель) КУ-1 в більшій мірі і в більш широкому діапазоні спектру зберігає прозорість після нейтронного, електронного та гамма-опромінення. Другий тип кварцу, КС-4В, з відносно низьким вмістом хлору, виявляється більш радіаційно-стійким в УФ-області спектру.

## ИССЛЕДОВАНИЕ РАДИАЦИОННОЙ СТОЙКОСТИ КВАРЦЕВЫХ СТЕКОЛ К НЕЙТРОННОМУ И ГАММА-ОБЛУЧЕНИЮ

***Б.А. Левин, Д.В. Орлинский, К.Ю. Вуколов, В.Т. Грицына***

Описаны результаты исследований радиационной стойкости некоторых видов кварцевых окон для ожидаемых радиационных условий термоядерного реактора. Испытания нескольких выбранных материалов показали, что кварц КУ-1, имеющий высокое содержание гидроксила ОН, лучший из них для видимой области спектра. По сравнению с другими

испытанными образцами материалов (сапфир, цериевое стекло и шпинель) КУ-1 в большей степени и в более широкой области спектра сохраняет прозрачность после нейтронного, электронного и гамма-облучения. Другой тип кварца, КС-4В, с относительно низким содержанием хлора, оказался несколько более радиационно-стойким в УФ-области спектра.